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SCIENTIFIC REVIEW

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## Oxidative Degradation of Polyaromatic Hydrocarbons under Ultrasonic Impact

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Received August 18, 2023; revised August 22, 2023; accepted March 16, 2024

**Abstract**—Aromatic and, in peculiar, polyaromatic hydrocarbons are one of the common class of anthropogenic pollutants. Processes of their degradation are of great interest from an ecological point of view. This article describes the current situation in the use of ultrasound as an instrument, accelerating such degradation.

**Keywords:** environmental pollution, polyaromatic hydrocarbons, ultrasonic impact, degradation of organic species

**DOI:** 10.3103/S0027131424700445

### INTRODUCTION

Polyaromatic hydrocarbons (PAHs) have carcinogenic and mutagenic activity, and they are among the most dangerous environmental pollutants. PAHs are formed as a by-product during combustion of almost any organic substances, in particular motor fuels. In addition, PAHs are part of the wastewater from the paint, varnish, and textile industries, as well as oil refining plants. Therefore, the task of cleaning the environment from PAHs is relevant, many works have carried out research in this area, among which information on the use of ultrasound exposure on the process of oxidative destruction of PAHs is of particular interest.

The use of ultrasound to intensify chemical reactions in a solution is based on the phenomenon of acoustic cavitation [1]. Cavitation occurs when an ultrasonic field acts on a liquid, resulting in the alternate local compression and expansion of the liquid, resulting in the formation of oscillating microbubbles. The bubbles grow, reaching a critical size, after which they collapse [2]. The size of a cavitation bubble can reach 100–200  $\mu\text{m}$  and its lifetime is several microseconds [3]. At the moment of the collapse of cavitation bubbles, local areas with a pressure of about 2000 atm and a temperature of up to 5000°C are formed. Various mechanochemical processes can occur in these areas [4, 5]. The propagation of a high-intensity ultrasonic wave with subsequent cavitation leads to the generation of highly reactive radicals:  $\text{H}^\bullet$ ,  $\text{OH}^\bullet$ ,  $\text{O}^\bullet$ , and  $\text{HO}_2^\bullet$  [6]. These radicals, interacting with organic substances contained in the solution, cause the degradation of the latter. The ultrasound frequency at which

the cavitation process is recorded lies in the range from 20 to 1000 kHz.

Before discussing the effect of ultrasound on the processes of oxidative degradation of PAHs, it makes sense to briefly consider the oxidation of lower aromatic hydrocarbons in an ultrasonic field using several examples.

The authors of the work [7] studied the kinetics of the oxidative degradation of benzene, ethylbenzene, and styrene in aqueous solutions (concentrations of about 0.1–1.0 mM) under the influence of ultrasound with a frequency of 520 kHz. It was shown that the reaction is of the first order with respect to the hydrocarbon. An attempt to correlate the Henry constants and the parameters of the kinetic equation showed that the degree of correlation is low. Similar results were obtained in [8] (oxidized hydrocarbons: naphthalene and phenanthrene). It was found that the rate constant of oxidative degradation increases with the increasing pH of the solution. As the temperature increases to 50°C, the reaction rate increases, then begins to decrease. The authors do not explain the nature of such a dependence.

The work [9] reports on the oxidation of various aromatic hydrocarbons under the influence of ultrasound. Naphthalene is converted into phthalic acid, anthracene into anthraquinone, and stilbene and styrene into benzoic acid. The authors used potassium periodate as an oxidizing agent, and the  $\text{K}_2\text{RuCl}_5$  complex, present in catalytic quantities, as an oxygen carrier. Under the influence of periodate and ultrasound, this complex is converted into a mixture of ruthenium tetroxide and  $\text{K}_3(\text{RuO}_3(\text{OH})_2)$ , which is reduced by hydrocarbons to the initial pentachlororu-

thenate. The yields of oxidation products range from 75% (in the case of phthalic acid) to 94% (in the case of benzoic acid). It is noted that high yields require the presence of acetonitrile in the system, whose role in the reaction is not completely clear. It is assumed that acetonitrile stabilizes the transition oxidation states of ruthenium.

The article [10] studies aromatic bromination in the  $\text{KBr-H}_2\text{O-CCl}_4$  system under the influence of ultrasound. It was found that for toluene, as well as *m*- and *p*-xylene, bromination mainly occurs in the benzyl position, no dibromination products were detected, and the total yield of bromine derivatives ranges from 55 to 95%. In pure carbon tetrachloride, the reaction does not occur. In the absence of potassium bromide, the chlorination reaction occurs, but its yields do not exceed 40%. The key stage of the process is water homolysis, and carbon tetrachloride becomes a trap for hydrogen atoms, preventing them from recombining with hydroxyl radicals.

Thus, the lower aromatic hydrocarbons can undergo oxidative transformations under the influence of ultrasound, forming nontoxic compounds (in the limit, of carbon dioxide and water).

The problem of neutralization of PAHs (as significantly more dangerous compounds) has attracted considerable attention of researchers. In the work [11], the oxidative degradation of PAHs catalyzed by ruthenium compounds, accelerated by ultrasound exposure, was studied. Pyrene, chrysene, diphenyl, naphthalene, anthracene, and phenanthrene were used as substrates. Oxidation was carried out in a mixture of water, acetonitrile, and methylene chloride, the oxidizer was sodium periodate, and the catalyst was ruthenium trichloride. The authors note that ultrasound sharply reduces the time required for oxidation: it decreases from 12 to 1.5–2 h. Oxidation of PAHs in most cases leads to products of the quinoid structure. It was shown that in the studied system ruthenium tetroxide is formed in situ, which is the direct oxidizer. The role of sodium periodate is reduced to the reoxidation of ruthenium.

The authors [12] studied the degradation process of PAHs (16 compounds) contained in textile wastewater sludge. The use of a composition of iron powder and EDTA, subjected to ultrasound exposure in air and representing an analog of the Fenton system, leads to the formation of hydrogen peroxide. At the same time, it is known [13] that organic substances degrade effectively in the Fenton system.

It has been shown [12] that the iron salts required for the Fenton system to function can be formed from metallic iron powder in the presence of EDTA both under the influence of ultrasound (frequency 20 kHz) and without it. However, in the case of ultrasound exposure, the maximum achievable concentration of iron in the solution is approximately twice as high as in its absence. The concentration of divalent iron and the

total concentration of iron in the solution depend on time in an extreme manner: a maximum is observed on the curve of this dependence. The degree of degradation increases with an increase in the EDTA concentration from 0 to 2 mM and then reaches a plateau. A similar dependence is observed for iron powder in the system, reaching a plateau corresponding to 15 g/L (EDTA concentration 0.2 mM). After 60 min of the reaction, the concentration of PAHs in the sludge drops by 40–50% and it decreases slightly as the molecular weight of PAHs increases. Ultrasound exposure with a power of 1.08 W/mL increases this figure to 70–80%. A decrease in power leads to a decrease in the degree of degradation, while a further increase practically does not change the situation.

The authors of the work [12] studied the pH dependence of the degree of degradation at the optimal values of other parameters. It was found that degradation occurs best in a slightly acidic environment (pH 5–6), a decrease in pH to 3 leads to a slight decrease in the degree of degradation, and an increase to 9 leads to a sharp drop. The latter fact is obviously associated with the removal of iron compounds from the reaction medium in the form of hydroxides.

A similar study was conducted in [14]. It also studied the degradation of PAHs from textile waste sludge. The oxidation system used by the authors of [14] is the same as that used in [12]. However, in [14] it was found that the presence of silicate and hydrophosphate ions in the system significantly increases the rate of PAH degradation.

A similar study was performed by the authors in [15]. In this case, PAHs from textile mill wastewater sludge were oxidized using the traditional Fenton reagent both with and without ultrasound exposure. It was found that in the absence of the Fenton reagent, the maximum degradation under ultrasound exposure (20 kHz) for naphthalene and tricyclic PAHs was achieved at an ultrasound power of 1.44 W/mL, and for heavier PAHs, at 1.80 W/mL. A further increase in power did not lead to an increase in the degree of degradation. The best results (about 65%) at pH 3 and 25°C for 30 min were achieved for naphthalene and pentacyclic hydrocarbons, while for tri-, tetra-, and hexacyclic hydrocarbons, it was approximately 35–40%.

In the case of using the Fenton reagent without ultrasound exposure, the degradation process also occurs. The maximum values of the degradation degree are achieved with the simultaneous action of ultrasound and the Fenton reagent; after 30 min it ranges from 75 to almost 100%. In all cases, naphthalene and pentacyclic PAHs are removed most effectively, while tetracyclic PAHs are removed the worst. The  $\text{Fe}^{2+} : \text{H}_2\text{O}_2$  ratio plays an important role in this. For PAHs with different numbers of cycles, the optimal value ranges from 1 : 3 to 1 : 1; exceeding this range does not lead to an increase in the degradation degree, and sometimes leads to a noticeable decrease.

The degradation degree in this case ranges from 65 to 90%. The concentration of the Fenton reagent is also important. The best results are achieved at a concentration of 140 mM; its further increase does not lead to an increase in the degradation efficiency.

The work [16] studies the influence of various factors (temperature, concentration, frequency and power of ultrasound, presence of foreign substances) on the process of the ultrasonic degradation of naphthalene, acenaphthylene, and phenanthrene. It was found that at the initial concentration of each hydrocarbon of 50  $\mu\text{g/L}$  (total concentration 150  $\mu\text{g/L}$ ), almost complete degradation occurs after 30 min of ultrasound exposure (80 kHz, 150 W, 20°C). For the initial concentration of 100  $\mu\text{g/L}$  (total 300  $\mu\text{g/L}$ ), this requires 60 min. Reducing the power to 75 W reduces the degree of degradation of naphthalene to 92%, while phenanthrene and acenaphthylene degrade completely at this power. At a power of 45 W, after 60 min, naphthalene and phenanthrene degrade by approximately 50%, acenaphthylene by 75%. A decrease in the ultrasound frequency also leads to a decrease in the degree of degradation. Thus, the degree of degradation of phenanthrene and naphthalene at 45 kHz, 150 W, and 20°C after 60 min of ultrasound exposure is 83–84%; and acenaphthylene, 96%. A decrease in power in this case also leads to a decrease in the degree of degradation. An increase in the temperature of the reaction medium leads to a slight decrease in the degradation rate; the authors associate this effect with the peculiarities of the formation of cavitation cavities. The addition of *n*-butanol, which is capable of reacting with hydroxyl radicals, to the reaction medium in a concentration approximately corresponding to the total concentration of PAHs, reduces the degradation rate. The presence of divalent iron ions in the system also has a significant effect on the PAH degradation rate. If their concentration is comparable to the concentration of PAHs, a sharp increase in the degradation rate is observed. The authors explain this by the fact that hydrogen peroxide, which is obtained during the recombination of hydroxyl radicals in the presence of  $\text{Fe}^{2+}$  ions, forms a Fenton system, in which the oxidation of organic substrates is known to occur effectively. At the same time, at a concentration of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  of 100 g/L, the degradation rate is significantly lower than in their absence. Probably, iron ions compete with PAHs for hydroxyl radicals. An interesting effect is observed when sodium chloride (10%) is introduced in the system. In this case, the degradation reaction has a noticeable induction period. The authors also associate this effect with the peculiarities of the formation of cavitation cavities (due to a change in surface tension). The effect of the ultrasound frequency on the degradation of individual PAHs (naphthalene, phenanthrene, anthracene, pyrene, and benzo[*k*]fluoranthene) in aqueous solutions was studied in [17]. It was found that the rate constant of the degradation process was

higher in the case of high-frequency ultrasound exposure (506 kHz). It also depended on the saturated vapor pressure, solubility in water, and the Henry constant of the corresponding hydrocarbon. As these parameters increased, the degradation rate constant increased. The authors of [18] believed that this dependence was general. In all cases, the degradation rate decreased in the following series: naphthalene—phenanthrene—anthracene—pyrene—benzofluoranthene. Nevertheless, at an ultrasound frequency of 506 kHz and a temperature of 20°C, 100% degradation of all the PAHs studied, except for benzofluoranthene, was achieved in 40 min. For the latter, the degree of degradation was about 60%. At an ultrasonic frequency of 20 kHz under similar conditions, complete degradation can only be achieved for naphthalene. The degree of degradation of phenanthrene, anthracene, pyrene, and benzofluoranthene in this case is approximately 80, 75, 60, and 25%, respectively. The author [17] associates the increase in the degradation efficiency with the increase in the number of cavitation cavities in the solution per unit time. In this work, it was found that the main degradation product of the studied PAHs is carbon monoxide, with carbon dioxide formed in significantly smaller quantities. In addition, acetylene and methane are also formed in approximately equal (but smaller compared to carbon dioxide) quantities. According to the author, this indicates the pyrolytic decomposition of PAHs in the collapsing cavitation cavities with the subsequent oxidation of the pyrolysis products by the hydroxyl radicals formed there. However, the article [19] proposes a mechanism that does not include the pyrolysis stage and implies only the sequential interaction of PAHs with hydroxyl radicals. This is supported by the detection of hydroxylated derivatives of PAHs in the degradation products [19].

The article [20] also studies the degradation rate of naphthalene, phenanthrene, and pyrene in aqueous solutions depending on the ultrasound frequency. It is shown that phenanthrene degrades most rapidly, and the dependence of the degradation rate on frequency is extreme for all three hydrocarbons (optimal frequency 582 kHz).

The effect of pH on the process of PAH degradation under the action of ultrasound is the subject of the work [21]. Air was used as an oxidizer. It was shown that a decrease in pH leads to an increase in the rate of the PAH degradation; the authors associate this with a change in the value of the oxidation—reduction potential of the hydroxyl radicals formed in the reaction medium under the action of ultrasound. Thus, at pH 2 this value is 2.7 V; and at pH 7, 1.8 V. It was also found that in aqueous-organic media the degradation rate decreases as the content of the organic component increases.

The effect of organic impurities on the ultrasonic degradation of PAHs in aqueous solutions was studied in [22]. It was found that in most cases the presence of

organic impurities in water sharply reduces the rate of PAH degradation (usually by factors of 2–5). The impurities of benzoic and humic acids, as well as pentanol, inhibit the reaction most strongly (at the same concentration). Pentane impurity changes the degradation rate significantly less, and in some cases it even increases. The effect of the degree of water oxygenation was also studied. It was shown that preliminarily blowing oxygen through the studied solution increases the oxidation rate by one-and-a-half to two times.

A similar study was undertaken by the authors in [23]. Its subject was the degradation of PAHs (anthracene, phenanthrene, pyrene) in pure water, as well as in the presence of foreign organic substances (benzoic acid, fulvic acids) under the action of ultrasound. The ultrasound power was 60 W and the frequency was 20 kHz (ultrasonic bath). It was found that in pure water phenanthrene ( $10^{-7}$  M solution) completely degraded after 5 min of exposure. It was found that in pure water the degradation rate constants for phenanthrene and pyrene were approximately the same, and for anthracene the constant was approximately 2.5 times higher. The presence of fulvic acids at a concentration of 20 mg/L reduced these values by a factor of 3.5 for anthracene and phenanthrene and by a factor of 2.5 for pyrene. Benzoic acid acts in a similar manner. In natural waters the degradation process can be completely suppressed. The most probable mechanism for this may be the obstruction of PAH access to cavitation cavities. It is noted that the primary particle formed from PAH is probably a cation-radical.

The dependence of the total amount of organic matter in sludge on the duration of treatment was studied in [15]. During the first 5 min, this amount increases from 32 to 41–44%. The authors explain this by the oxidation of organic matter contained in the sludge, an increase in the amount of carboxyl groups in it, and the subsequent interaction of these groups with the aluminum and iron oxides contained in the sludge. In this case, organic salts of these elements are formed. Then the total content of organic matter begins to fall, but its decrease is small and does not exceed 10% of the value achieved after 5 min (the maximum exposure time is 60 min). In the article [24], however, it is noted that ultrasonic treatment of industrial waste sludge in some cases can lead to an increase in the concentration of PAHs in the solution. The authors associate this phenomenon with the desorption of PAHs firmly adsorbed on sludge particles under the action of ultrasound.

When using ultrasound to purify wastewater from PAHs, oxidizers such as ozone [25] and hydrogen peroxide can be used, both in pure form [26] and as part of Fenton's reagent [27]. Naturally, the process using ultrasound without additional reagents is preferable [28, 29].

In [30], a model system of 16 PAHs dissolved in water (total concentration of 0.1 ppm) was studied; the

experiments were carried out in air. Different ranges of emitted energy and ultrasound frequency were studied. Thus, with an emitted energy of 4 J/mL, light PAHs (up to and including pyrene) are decomposed by not more than 70–75%. The degree of degradation of heavier PAHs is higher; for all the PAHs studied, it is up to 90%. The authors showed that an increase in the power of exposure does not always lead to an increase in the degree of degradation.

The authors of [31] used the method of solid-phase extraction in combination with chromatograph mass spectrometry to study the degradation of eight PAHs in aqueous solutions under the action of ultrasound. The concentration of PAHs varied within the range from 0.1 to 50 µg/L. All the experiments were carried out in air. It was found that with continuous sonication (80 kHz, 75 W of electric power) at least 90% degradation of the studied hydrocarbons could be achieved in 3 h. Anthracene and acenaphthylene decompose better than others, and pyrene worse, but the difference is small. Under thermostating conditions (25°C), the degradation of anthracene and acenaphthylene in 3 h reaches 98–99%, and that of pyrene, ~90%. In the absence of thermostating, the degradation in 3 h is almost 100% for all the studied PAHs. In the 50% duty cycle mode (1 min of sounding, 1 min without sounding), the picture becomes significantly more complex both with and without thermostating.

In the work [32], which also studies the ultrasonic degradation of PAHs in aqueous solutions, the variable parameters were the sonication time and the amplitude of ultrasonic vibrations. The highest degree of degradation (76%) was achieved at the maximum amplitude (114 µm), but the experiment time in this case was not specified. Further experiments were carried out at this amplitude with the maximum sonication time of 45 min. The degree of degradation increased with time, reaching a value of 59% after 45 min. If the system is ozonized under the same conditions, the degree of degradation increases to 96%; in the absence of ultrasound, similar ozonation gives only 73% degradation.

The article [33] describes the neutralization of petrochemical waste using anaerobic bacteria that produce biogas. However, PAHs strongly inhibit bacterial growth; thus, the authors consider the possibility of preliminary PAH degradation under the action of either thermal (130°C) or ultrasonic (20 kHz, 480 W) treatment for 20 min. It is noted that ultrasound causes PAH degradation, but thermal treatment is more effective.

The authors in [34] consider the process of chrysene extraction from river silt using butylthiopheny bromide. It was found that with ultrasound exposure, extraction occurs faster, but both the extractant and chrysene are subject to destruction.

In the article [35], the degradation of phenanthrene and pyrene adsorbed on river silt particles in an

aqueous suspension under the influence of ultrasound was studied. The ultrasound frequency was 20 kHz and the power was 430 W/L. It was shown that the particle size affects the degradation rate: for large sorbent particles it is higher. Similarly, the bioavailability of the resulting products depends on the particle size. Increasing the suspension concentration reduces the degradation rate.

The authors of [36] investigated the effect of the ultrasonic treatment of contaminated river sediments on the biodegradation of PAH degradation products (naphthalene, phenanthrene, and pyrene). The ultrasound frequency was 20 kHz at a power of 430 W. Depending on the sample, the concentration of the aforementioned PAHs ranged from 0 to 560 nmol/g with a total organic carbon content of 2 to 16% (all figures refer to dry samples). It was found that the total amount of these PAHs in the sludge decreased after ultrasonic treatment. Phenanthrene degraded the most, while naphthalene degraded the least. However, the degree of degradation was low and did not exceed 25% after 80 min of treatment.

The dynamics of the accumulation of the biodegradable fraction of PAH degradation products were also of considerable interest. It was shown that the amount of these products usually increases after the start of ultrasonic treatment, but quickly reaches a plateau (within 20 min). The data are related only to phenanthrene and pyrene; for naphthalene, the measurement error is excessively large (about 40%). In general, the method used by the authors does not seem effective. A similar study was carried out by the author of [37]; it was shown that adsorbed PAHs degrade worse than dissolved ones, and ultrasonic treatment causes the desorption of PAHs from sludge particles.

The authors of [38] studied the ultrasonic degradation of PAHs in wastewater from oil refineries. It was found that the degradation rate increased with the increasing NaCl concentration in solutions. With an increase in the sodium chloride concentration from 1.5 to 12 g/L, the degree of PAH degradation after 150-min ultrasonic treatment increased from 72–78 to 97–99%. It was noted that light PAHs degrade mainly due to interaction with hydroxyl radicals, while heavy ones are mainly subject to pyrolysis in collapsing cavitation cavities. In [39], the same objects were studied, and the dependence of the degradation degree on temperature and the concentration of oxidizing agents (dissolved oxygen, hydrogen peroxide) was studied. An increase in temperature and the presence of oxidizing agents contribute to an increase in the degree of degradation. Degradation occurs most effectively in the presence of hydrogen peroxide.

A similar study was described by the same authors in [40]. The difference in its content from the two previous ones is that the degradation of aqueous solutions of PAHs in some cases occurred in the presence of carbon tetrachloride (200–1000 mg/L). This compound

can form active radicals ( $\text{Cl}^\bullet$ ,  $\text{CCl}_3^\bullet$ ) and dichlorocarbene when exposed to ultrasound. These particles are capable of reacting with PAHs, causing their chemical transformations. It was shown that the presence of  $\text{CCl}_4$  in the system in an amount of 600 mg/L (optimal concentration) can increase the degree of degradation of benzo[*k*]fluoranthene and benzo[*a*]pyrene from 80 to 97% (60°C, 150 min of ultrasound exposure). The authors also found that the resulting products are non-toxic for *Daphnia magna*.

Thus, the published data indicate that ultrasound is a useful tool for increasing the efficiency of the degradation of aromatic hydrocarbons, especially polyaromatic ones. Ultrasound can be used to clean even such complex objects as sludge contaminated with PAHs.

It is difficult to draw unambiguous conclusions about the influence of various parameters on the efficiency of destruction based on the available data. However, certain patterns can be observed. An increase in ultrasound power in most cases leads to an increase in the degree of degradation with a subsequent plateau. The dependence of the degree of degradation on the ultrasound frequency is extreme, the optimum being in the range of 500 to 600 kHz. The presence of foreign substances usually affects the process. The presence of iron ions has a beneficial effect due to the formation of a Fenton-like system. Nonaromatic organics, on the contrary, inhibit degradation, probably due to competitive oxidation.

It should also be borne in mind that the use of more efficient and economical ultrasonic action allows eliminating the mechanical mixing of reagents.

#### AUTHOR CONTRIBUTION

All authors made an equivalent contribution to the preparation of the publication.

#### FUNDING

This study was carried out as part of a state task of the Department of Chemistry of Moscow State University "Petrochemistry and catalysis. Rational use of carbon-containing raw materials," CITIS no. 121031300092-6.

#### ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This work does not contain any studies involving human and animal subjects.

#### CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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Translated by M. Drozdova

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